

PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
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PERIOD COVERED: February, 1988

I. SIDESTREAM SMOKE

- A. Objective:** Conduct analyses on mainstream and sidestream smoke to determine qualitative and quantitative differences between control and reduced visibility cigarettes. Develop methods to analyze for selected components. Demonstrate effective methods for reduction or removal of these components.
- B. Status:**
1. Sidestream gas phase analysis is now being routinely carried out with a 60 M, 1 micron film thickness DB-5 column. Sidestream gas phase from cigarettes prepared with 35% $\text{Mg}(\text{OH})_2$ and 35% CaCO_3 wrappers has been compared with that from Marlboro Lights as control. The effluent was monitored by FID and NPD and the data examined using the PE LIMS system. Gas phase composition was studied by MS by direct syringe injection of 50 ml samples.
 2. Several different samples of sidestream smoke have been analyzed using the silica-gel trapping technique.
 - a. The sidestream smoke from a cigar was collected and worked up in the usual fashion.
 - b. Sidestream from Marlboro Lights was collected under both static and dynamic (puffing) conditions.
 - c. Samples of the 35% $\text{Mg}(\text{OH})_2$ and 35% CaCO_3 wrapped cigarettes were studied vs Marlboro Lights as control.
 - d. RL baseweb samples prepared individually from bright, burley and oriental were obtained and smoked. An LTF model was also smoked in this series. These essentially "cellulose only fillers" were chosen to compare the ethyl acetate fractions with those of "normal" blends.
- C. Results:**
1. Good reproducibility is obtained for the gas phase analyses except for the early, low molecular weight compounds which are trapped unreproducibly in the liquid-nitrogen cooled trap at the head of the analytical column. The samples examined produced very similar profiles with both NPD and FID detectors. The gas phase from Parliament Lights 100's was studied for compound identification. Fifty-five components ranging from carbon dioxide to limonene have been determined to date. These identifications will be useful for establishing elution order on non-polar columns and for comparison with whole smoke work carried out with the Finnegan MS.
 2. A few new compounds have been identified by MSD. The list of compounds identified to date has been consolidated, revised and additional and corrected CAS numbers added.
 - a. No significant differences were seen between cigar smoke, control cigarette smoke and low ss cigarette smoke.
 - b. No significant differences were observed when ss smoke was collected from statically or dynamically burned models.

- c. Small quantitative differences were observed between the low sidestream models utilizing different wrappers.
- d. The FID profiles of the baseweb samples vs normal blends were surprisingly similar. However, examination by MSD revealed significant qualitative differences. The baseweb samples were very similar to each other but appear to contain more cellulosic degradation products than do the normal blend samples.

- D. **Conclusions:** 1. Significant progress has been made in improving gas phase chromatographic resolution. However, as seen previously, few differences are seen between the gas phase profiles of various cigarette types.
2. Few differences have been observed in the sidestream smoke between the various models studied utilizing the silica gel collection technique. However, striking differences have been observed between the ethyl acetate fractions of the sidestream and mainstream smoke from the same sample with this method. This indicates that the method is capable of discerning differences if they are there to be seen and that the similarity of sidestream samples is simply a reflection of the true nature of the samples. That is, the chemical composition of sidestream or mainstream smokes from different tobaccos is in fact very similar.
- E. **Plans:** Continue GC/MS studies on the mainstream and sidestream smoke from cigarettes smoked under both static and dynamic conditions. Generate data from multiple runs to determine qualitative and quantitative mainstream and sidestream gas phase comparisons.

II. Miscellaneous

- A. **Objective:** 1. Upgrade computer capabilities for the Project and for the Division as required.
2. Conduct sidestream smoke visibility measurements as required.
3. Determine experimental conditions necessary to support the program to incorporate selected ingredients into cigarette models to improve sidestream smoke properties.
4. Conduct GC/MS and pyrolysis/GC/MS analyses as required.
- B. **Results:** 1. The upgrade to the P.E. system has been delivered, installed and is operational. A file transfer protocol is available to allow direct transfer from the Perkin-Elmer system to the UNIX system. The 850Mb disk drive has been installed and is currently undergoing testing.
2. Several sets of cigarettes have been studied for visibility and related data. These include those to determine the effect of CaCO_3 wrappers as compared to the $\text{Mg}(\text{OH})_2$ models.

3. Procedures are being studied to conduct these analyses utilizing GC and GC/MS.

4. Several requested analyses were carried out.

C. Conclusions: 1. Greatly expanded file transfer and data manipulation capability is now possible through the upgraded systems.

2. Some differences exist between the visibilities determined for CaCO_3 wrapped cigarettes when compared to $\text{Mg}(\text{OH})_2$ models. The new small particle size CaCO_3 papers appear to give less reduction in visible sidestream smoke but apparently have positive subjective properties when compared to the $\text{Mg}(\text{OH})_2$ models of comparable composition.

3. Methods will shortly be available to quantitatively determine the detected amounts of vanillin found in the MS TPM, SS TPM, SS chamber wash and butt from cigarettes to which known amounts of vanillin have been applied.

4. Vanillin was the only detected component found in the 300 deg C pyrolysis of CR-2673 (glucovanillin). At 600 deg, vanillin (63%), catechol (27%), unknown m/z 150 (6%) and hydroxybenzaldehyde (3%) were detected. No impurities were found in the 300 and 600 deg C pyrolyses of Darco-G charcoal.

D. Plans: 1. Continue upgrades to the current systems as they become available. Continue to expand the RS1 data files as new smoke components are identified. Enhance system management capability by a project members attendance at the Perkin-Elmer system managers course.

2. Continue to study cigarette models for visibility and related information as they become available.

3. Continue to investigate the distribution and quantitation of selected ingredients applied to cigarette models to enhance sidestream subjectives.

4. Supply GC and GC/MS analyses as required.